PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 21 February 2001 (21.02.01)	
International application No. PCT/SE00/01435	**Applicant's or agent's file reference** 0004 PCT
International filing date (day/month/year) 05 July 2000 (05.07.00)	**Priority date** (day/month/year) 06 July 1999 (06.07.99)
Applicant DAHLLÖF, Håkan et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

12 January 2001 (12.01.01)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland** Facsimile No.: (41-22) 740.14.35	Authorized officer F. Baechler Telephone No.: (41-22) 338.83.38

14

PATENT COOPERATION TREATY

PCT

REC'D 31 OCT 2001

WIPO PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
0004 PCT		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/SE00/01435	05.07.2000	06.07.1999

International Patent Classification (IPC) or national classification and IPC7

D 21 C 9/147

Applicant

Kvaerner Pulping AB et al

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
12.01.2000	19.10.2001
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Telex 17978 PATOREG-S Facsimile No. 08-667 72 88	Authorized officer Marianne Bratsberg/ELY Telephone No. 08-782 25 00

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/SE00/01435

I. Basis of the report

1. With regard to the **elements** of the international application:*

[ ] the international application as originally filed

[X] the description:
pages 1-9, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

[X] the claims:
pages ______, as originally filed
pages ______, as amended (together with any statement) under article 19
pages ______, filed with the demand
pages 13-16, filed with the letter of 13.08.2001

[X] the drawings:
pages 1-2, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

[ ] the sequence listing part of the description:
pages ______, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language English which is:

[ ] the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

[X] the language of publication of the international application (under Rule 48.3(b)).

[ ] the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

[ ] contained in the international application in written form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readable form.

[ ] The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. [ ] The amendments have resulted in the cancellation of:

[ ] the description, pages ______

[ ] the claims, Nos. ______

[ ] the drawings, sheet/fig ______

5. [ ] This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/SE00/01435

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-9	YES
	Claims		NO
Inventive step (IS)	Claims	1-9	YES
	Claims		NO
Industrial applicability (IA)	Claims	1-9	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

Amended claims have been filed with the letter of 13 August 2001.

Closest prior art cited in the International Search Report:

D1: WO 9715715 A1

D2: WO 9630586 A1

Document D1 discloses an arrangement for the oxygen delignification of pulp in two stages, comprising a first pump (1), a first mixer (2), a first delignification zone (3), a second pump (4), a second mixer (5) and a second delignification zone (6), see fig 1. Steam is admixed with the pulp in the second mixer. The pressure in the first stage is higher than the pressure in the second stage.

The system defined in the amended claim 1 differs from the arrangement in D1 in that an additional mixer (8) is arranged to receive pulp from the first delignification zone, i. e. prior to the second pump, which mixer comprises means for admixing steam with the pulp and in that the second pump has a pumping effect such that a lower oxygen pressure is obtained in the first delignification zone as compared with the second delignification zone.

.../...

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V.

Document D2 discloses a process and an arrangement for increasing the temperature of a pulp suspension transported to a bleaching treatment under pressure with steam. A mixer is arranged prior to the pump that increases the pressure of the suspension, which mixer comprises means for admixing steam with the pulp. The purpose of this process is to be able to use low-pressure steam as heating means. However, this document does not disclose a system for the oxygen delignification of pulp in two stages, in which the second stage is performed at a higher pressure than the first stage.

Thus, it is not considered to be obvious to a person skilled in the art to combine the teachings from D1 and D2 so as to arrive at the system defined in claim 1.

Therefore, the invention defined in claim 1 is novel and considered to involve an inventive step.

The process defined in claim 9 is novel and considered to involve an inventive step for the same reasons as for claim 1.

PATENT CLAIMS

1. System for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, which oxygen delignification takes place in at least two stages and where the system is
c h a r a c t e r i z e d i n that it comprises,
- a first pump (1) which is arranged to pump the pulp to a first mixer (3) for admixing chemicals which are required for the oxygen delignification in this first mixer (3), which first mixer is arranged in close conjunction with the first pump,
- a first delignification zone (6) which is arranged to receive pulp from the first mixer (3),
- a second mixer (8) which is arranged to receive pulp from the first delignification zone, which second mixer comprises means for admixing steam (MP steam) with the pulp,
- a second pump (4) which is arranged to receive pulp after the second mixer,
- a third mixer (5) which is arranged in close conjunction with the second pump, for admixing chemicals which are required for the oxygen delignification in this third mixer (5),
- a second delignification zone (10) which is arranged to receive the pulp from the third mixer.

2. System for oxygen delignification according to Claim 1,
c h a r a c t e r i z e d i n that the first and third mixers (3 and 5, respectively) are mixers using mechanical agitation, with the pulp at least partially being fluidized in gaps in the mixer, and in that the second mixer (8) is a static mixer without mechanical agitation.

3. System for oxygen delignification according to Claim 2,
c h a r a c t e r i z e d i n that it comprises means (O_2) for adding oxygen to the first (3) and third (5) mixers, respectively, and means (MP steam) for adding steam to the second mixer (8).

4. System for oxygen delignification according to Claim 3,
c h a r a c t e r i z e d i n that the second mixer comprises means for supplying steam in a controllable manner (7,V,TC), preferably feedback-controlled in dependence on the temperature of the pulp after the said mixer.

5. System for oxygen delignification according to Claim 4,
c h a r a c t e r i z e d i n that the second mixer (8) consists of a pulp-conveying pipe having a number of inlet holes for the steam in the wall of the pipe.

6. System for oxygen delignification according to Claim 5,
c h a r a c t e r i z e d i n that the steam consists of medium-pressure steam at 8-14 bar.

7. System for oxygen delignification according to Claim 3,
c h a r a c t e r i z e d i n that the system comprises a control system (PC) for controlling the rotational speed of the second pump (4) depending on the pressure in the first delignification zone (6).

8. System for oxygen delignification according to Claim 1,
c h a r a c t e r i z e d

- in that the first delignification zone (6) has a volume which results in a dwell time of 2-20 minutes, preferably 2-10 minutes, and even more advantageously 3-6 minutes, for the pulp in the first delignification zone,
- in that the system is adjusted such that the pressure in the first delignification zone amounts to 0-6 bar, preferably 0-4 bar,
- in that the second pump (4) has a pumping effect such that the pressure in the second delignification zone reaches a level of at least 3 bars overpressure at the top of the second delignification zone, and
- in that the second delignification zone (10) has a volume which is at least 10 times greater than the volume of the first delignification zone, i.e. has a volume which results in a dwell time of at least 20-200 minutes, preferably 20-100 minutes, and even more advantageously within the range 50-90 minutes.

9. Process for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, in at least two stages,

c h a r a c t e r i z e d

a) in that pulp at median concentration is pressurized, and
b) after that, chemicals, chiefly oxygen, are added for the oxygen delignification, such that oxygen delignification takes place in a first stage in which the pulp is treated for a relatively short time, corresponding to 2-20 minutes, preferably 2-10 minutes, and even more advantageously 3-6 minutes, under moderate overpressure within the interval 0-6 bar, preferably 0-4 bar, and at a moderate temperature in the range 85°C ± 10°C,
c) and in that the pulp, after the first stage, and with the pulp being at a median concentration, is

first mixed together with steam for the purpose of increasing the temperature,

d) after which there follows a first pressurization of the heated pulp,

e) and, after that, a second addition of chemicals, chiefly oxygen, for the oxygen delignification,

f) in order, in a concluding stage, to be treated for a longer time than in the first stage, i.e. for a time which is of the order of magnitude of 10 times longer than in the first stage, in the interval 2-200 minutes, preferably 20-100 minutes, and even more advantageously in the interval 50-90 minutes, with this stage taking place at an initial pressure within the interval 8-10 bar, corresponding to the pressure at the inlet of the reactor, but also at a higher temperature, preferably in the range 100°C ± 10°C, but preferably at least 5°C higher than the temperature in the first stage.

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PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No. PCT/SE 00 / 0 1 4 3 5

International Filing Date 0 5 -07- 2000

The Swedish Patent Office
PCT International Application

Name of receiving Office and "PCT International Application"

Applicant s or agent s file reference *(if desired) (12 characters maximum)* 0004 PCT

Box No. I TITLE OF INVENTION

System and method for oxygen delignification of pulp made from lignocellulosic material

Box No. II APPLICANT

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

Kvaerner Pulping AB
Box 1033
S-651 15 KARLSTAD
SWEDEN

☐ This person is also inventor.

Telephone No. +46 54 194600

Facsimile No. +46 54 142253

Teleprinter No.

State *(that is, country)* of nationality: SWEDEN

State *(that is, country)* of residence: SWEDEN

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

DAHLLÖF Håkan
Torpstöd Samarkand
S-660 52 EDSVALLA
SWEDEN

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: SWEDEN

State *(that is, country)* of residence: SWEDEN

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is ~~hereby~~/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

FURHEM Hans
Kvaerner Pulping AB
Box 1033
S-651 15 KARLSTAD
SWEDEN

Telephone No. +46 54 194627

Facsimile No. +46 54 142253

Teleprinter No.

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III **FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)**

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

RAGNAR Martin
Malmtorgsgatan 1
S-653 40 KARLSTAD
SWEDEN

This person is:
- ☐ applicant only
- ☒ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: SWEDEN

State *(that is, country)* of residence: SWEDEN

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant s State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

Regional Patent

- [x] **AP** **ARIPO Patent:** **GH** Ghana, **GM** Gambia, **KE** Kenya, **LS** Lesotho, **MW** Malawi, **SD** Sudan, **SL** Sierra Leone, **SZ** Swaziland, **TZ** United Republic of Tanzania, **UG** Uganda, **ZW** Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- [x] **EA** **Eurasian Patent:** **AM** Armenia, **AZ** Azerbaijan, **BY** Belarus, **KG** Kyrgyzstan, **KZ** Kazakhstan, **MD** Republic of Moldova, **RU** Russian Federation, **TJ** Tajikistan, **TM** Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- [x] **EP** **European Patent:** **AT** Austria, **BE** Belgium, **CH and LI** Switzerland and Liechtenstein, **CY** Cyprus, **DE** Germany, **DK** Denmark, **ES** Spain, **FI** Finland, **FR** France, **GB** United Kingdom, **GR** Greece, **IE** Ireland, **IT** Italy, **LU** Luxembourg, **MC** Monaco, **NL** Netherlands, **PT** Portugal, **SE** Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- [x] **OA** **OAPI Patent:** **BF** Burkina Faso, **BJ** Benin, **CF** Central African Republic, **CG** Congo, **CI** Côte d Ivoire, **CM** Cameroon, **GA** Gabon, **GN** Guinea, **GW** Guinea-Bissau, **ML** Mali, **MR** Mauritania, **NE** Niger, **SN** Senegal, **TD** Chad, **TG** Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT *(if other kind of protection or treatment desired, specify on dotted line)* ....

National Patent *(if other kind of protection or treatment desired, specify on dotted line)*:

- [x] **AE** United Arab Emirates
- [x] **AL** Albania ....
- [x] **AM** Armenia ....
- [x] **AT** Austria ....
- [x] **AU** Australia ....
- [x] **AZ** Azerbaijan
- [x] **BA** Bosnia and Herzegovina ....
- [x] **BB** Barbados
- [x] **BG** Bulgaria ....
- [x] **BR** Brazil ....
- [x] **BY** Belarus ....
- [x] **CA** Canada
- [x] **CH and LI** Switzerland and Liechtenstein
- [x] **CN** China ....
- [x] **CR** Costa Rica ....
- [x] **CU** Cuba ....
- [x] **CZ** Czech Republic ....
- [x] **DE** Germany ....
- [x] **DK** Denmark ....
- [x] **DM** Dominica
- [x] **EE** Estonia ....
- [x] **ES** Spain ....
- [x] **FI** Finland ....
- [x] **GB** United Kingdom
- [x] **GD** Grenada
- [x] **GE** Georgia ....
- [x] **GH** Ghana ....
- [x] **GM** Gambia
- [x] **HR** Croatia ....
- [x] **HU** Hungary ....
- [x] **ID** Indonesia
- [x] **IL** Israel ....
- [x] **IN** India ....
- [x] **IS** Iceland
- [x] **JP** Japan ....
- [x] **KE** Kenya ....
- [x] **KG** Kyrgyzstan ....
- [x] **KP** Democratic People s Republic of Korea ....
- [x] **KR** Republic of Korea ....
- [x] **KZ** Kazakhstan ....
- [x] **LC** Saint Lucia
- [x] **LK** Sri Lanka
- [x] **LR** Liberia
- [x] **LS** Lesotho ....
- [x] **LT** Lithuania
- [x] **LU** Luxembourg
- [x] **LV** Latvia
- [x] **MA** Morocco ....
- [x] **MD** Republic of Moldova ....
- [x] **MG** Madagascar ....
- [x] **MK** The former Yugoslav Republic of Macedonia ....
- [x] **MN** Mongolia
- [x] **MW** Malawi ....
- [x] **MX** Mexico ....
- [x] **NO** Norway
- [x] **NZ** New Zealand ....
- [x] **PL** Poland ....
- [x] **PT** Portugal ....
- [x] **RO** Romania
- [x] **RU** Russian Federation ....
- [x] **SD** Sudan
- [x] **SE** Sweden
- [x] **SG** Singapore
- [x] **SI** Slovenia ....
- [x] **SK** Slovakia ....
- [x] **SL** Sierra Leone ....
- [x] **TJ** Tajikistan ....
- [x] **TM** Turkmenistan ....
- [x] **TR** Turkey ....
- [x] **TT** Trinidad and Tobago ....
- [x] **TZ** United Republic of Tanzania
- [x] **UA** Ukraine ....
- [x] **UG** Uganda ....
- [x] **US** United States of America ....
- [x] **UZ** Uzbekistan ....
- [x] **VN** Viet Nam ....
- [x] **YU** Yugoslavia ....
- [x] **ZA** South Africa ....
- [x] **ZW** Zimbabwe ....

Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

- [ ] ....
- [ ] ....

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. *(Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)*

PCT/SE 00 / 0 1 4 3 5

05 -07- 2000

Box No. VI PRIORITY CLAIM

☐ Further priority claims are indicated in the Supplemental Box.

Filing date of earlier application *(day/month/year)*	Number of earlier application	Where earlier application is: national application: country	regional application:* regional Office	international application: receiving Office
item (1) 6 July 1999 (06.07.1999)	9902586-8	SWEDEN		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office)* identified above as item(s): (1)

* *Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.*

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used)* :

ISA / SE

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority)*:

Date *(day/month/year)* Number Country *(or regional Office)*

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following **number of sheets**:

request	:	4 ✓
description (excluding sequence listing part)	:	9 ✓
claims	:	3 ✓
abstract	:	1 ✓
drawings	:	2 ✓
sequence listing part of description	:	
Total number of sheets	:	19 ✓

This international application is **accompanied by** the item(s) marked below:

1. ☐ fee calculation sheet
2. ☐ separate signed power of attorney
3. ☒ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
8. ☐ nucleotide and/or amino acid sequence listing in computer readable form
9. ☐ other *(specify)*:

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: Swedish

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Hans Furhem
Kvaerner Pulping AB

For receiving Office use only

1. Date of actual receipt of the purported international application:	05 -07- 2000
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:	
4. Date of timely receipt of the required corrections under PCT Article 11(2):	
5. International Searching Authority (if two or more are competent):	ISA / SE
6. ☐ Transmittal of search copy delayed until search fee is paid.	

2. Drawings:
☒ received:
☐ not received:

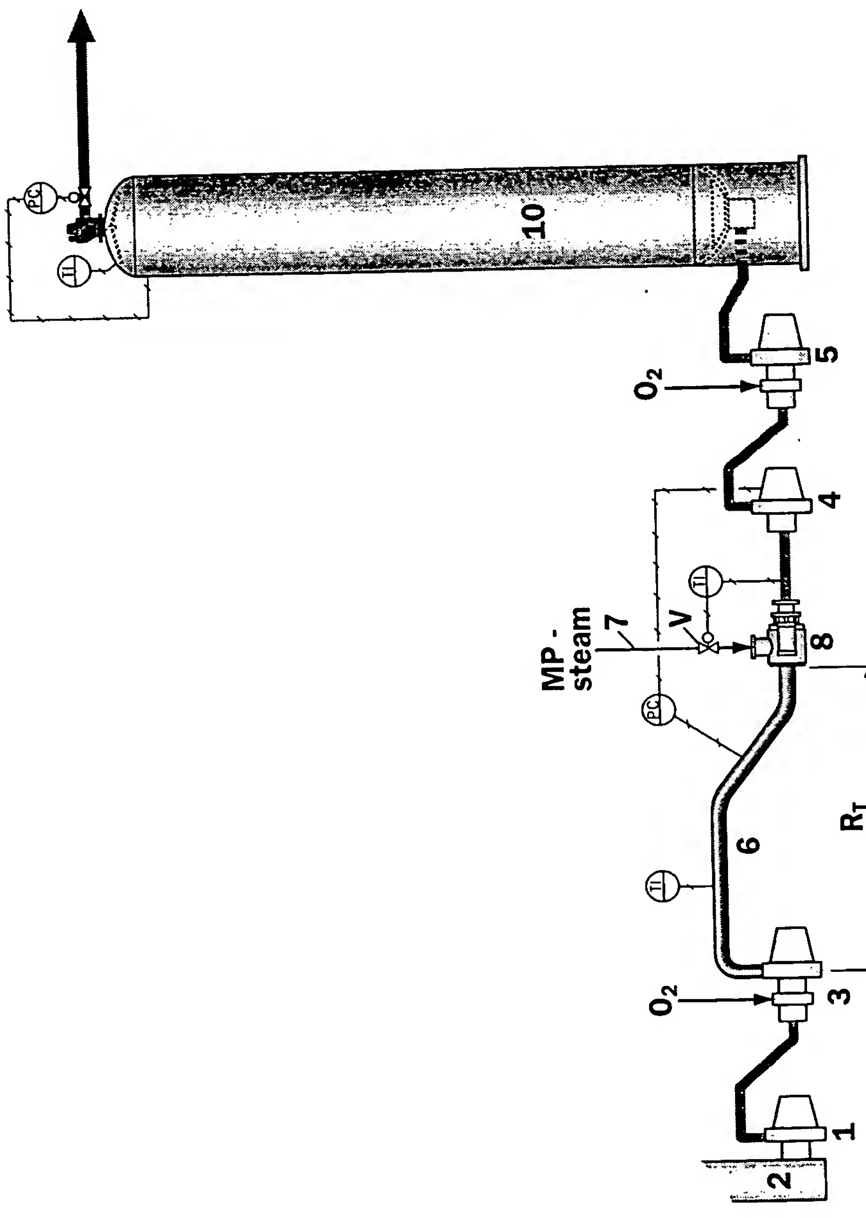
For International Bureau use only

Date of receipt of the record copy by the International Bureau: 04 A[illegible] 2000 04.08.00

Form PCT/RO/101 (last sheet) (July 1998; reprint January 2000)

See Notes to the request form

Fig1



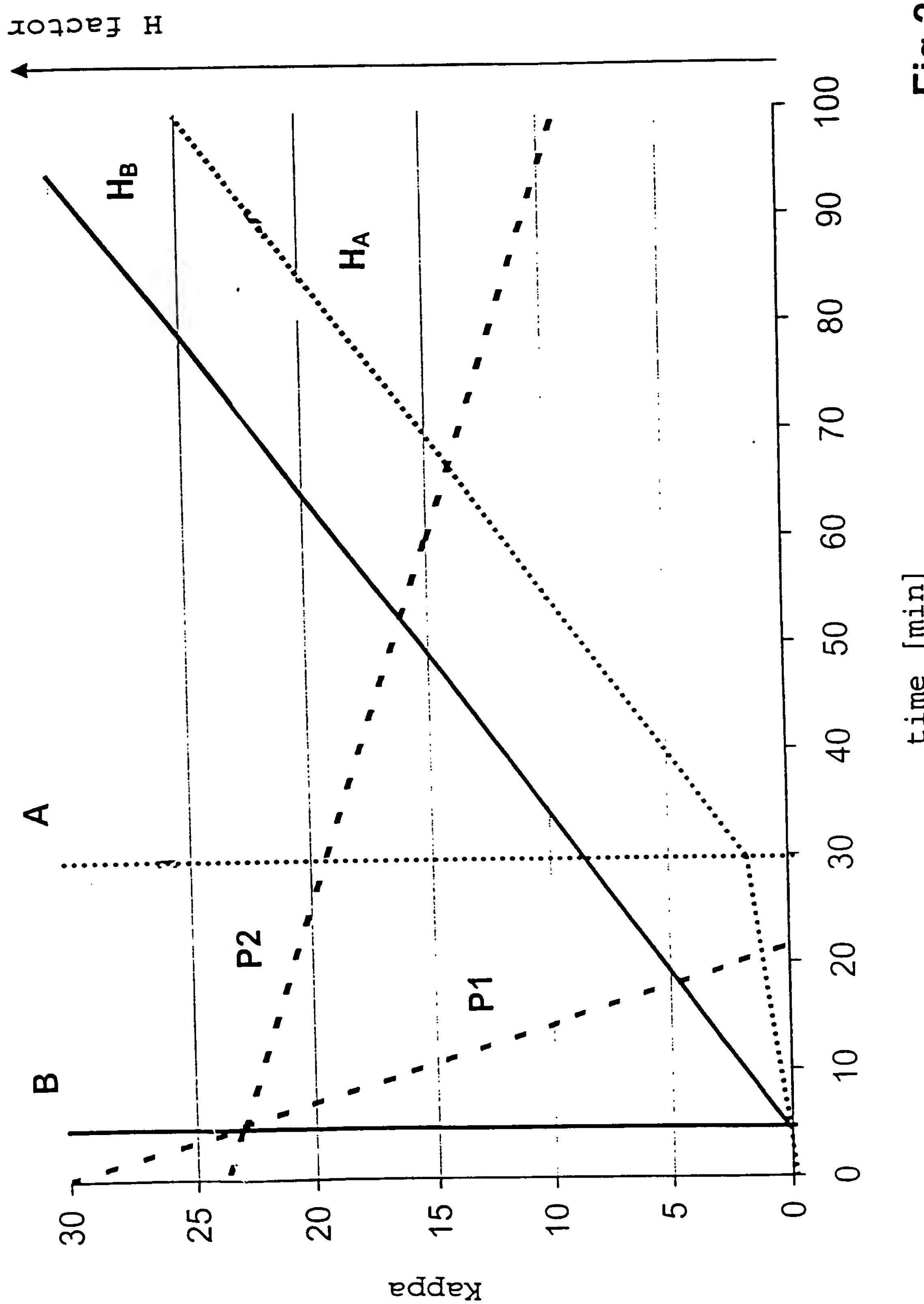

H factor
Kappa
time [min]
A
B
H_B
H_A
P1
P2
0
5
10
15
20
25
30
0
10
20
30
40
50
60
70
80
90
100

Fig.2

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System och förfarande för syrgasdelignifiering av massa av lignocellulosahaltigt material.

Föreliggande uppfinning avser ett system samt ett förfarande för syrgasdelignifiering i enlighet med ingressen till patentkravet 1 respektive 9.

Teknikens ståndpunkt

Ett flertal olika förfaranden för syrgasdelignifiering är kända.

Genom US,A,4.259.150 visas ett system med flerstegs syrgasblekning där man i varje steg först blandar upp massan till lägre konsistens med O_2, vatten samt NaOH, följt av en förtjockning tillbaka till den konsistensnivå som massan hade till det aktuella steget. Syftet är att få en ekonomisk klorfri blekning med högt utbyte. Samtidigt kan kappatalet genom upprepade steg sänkas från 70 ner till 15, eller till och med under 15.

Genom SE,C,467.582 visas ett förbättrat system för syrgasblekning av massa av medelkonsistens. Genom en optimerad temperaturkontroll sker en syrgasblekning i en första delignifieringszon med låg temperatur följt av en andra delignifieringszon med 20-40 graders högre temperatur. Syftet är att erhålla ett förbättrat utbyte och en förbättrad viskositet under bibehållen uppehållstid vid industriell tillämpning.

Utöver SE,C,467.582 har även andra varianter på syrgasdelignifiering i två steg patenterats. I SE,C,505.147 visas ett förfarande där massan i det första steget skall ha hög massakoncentration i området 25-40%, och i det andra steget 8-16%, samtidigt som temperaturen i det andra steget skall vara högre eller lika med temperaturen i det första steget, i linje med den temperaturskillnad som anvisas i SE,C,467.582. Fördelarna med lösningen enligt SE,C,505.147 anges vara möjligheter att blanda in mer syrgas i det första högkonsistensteget utan risk för kanalbildning, men där samtidigt icke utnyttjade syrgasmängder kan blödas av efter första steget för vidare inmixning i en andra mixer före det andra steget.

I SE,C,505.141 visas ytterligare ett förfarande, vilket är ett försök att kringgå SE,C,467.582, då det patentsökta anges vara att temperaturskillnaden mellan stegen ej

överstiger 20 grader, dvs. den undre lämpliga temperaturskillnaden patenterad i SE,C,467.582, men att temperaturskillnad ändock skall förefinnas. Därutöver anges att a) trycket skall vara högre i det första steget och b) att uppehållstiden är kort i första steget, i storleksordningen 10-30 minuter, samt c) uppehållstid i det andra steget längre, i storleksordningen 45-180 minuter.

Genom ett föredrag benämnt "*Two stage MC-oxygen delignification process and operating experience*" på *1992 Pan-Pacific Pulp & Paper Technology Conference('99 PAN-PAC PPTC), Sept. 8-10, Sheraton Grande Tokyo Bay Hotel & Towers*, hållet av Shinichiro Kondo från Technical Div. Technical Dept. OJI PAPER CO. Ltd, visas en lyckosam installation utförd 1986 med tvåstegs syrgasdelignifiering i en anläggning i Tomakomai.

I denna OJI PAPER-anläggning i Tomakomai matades massan med ett tryck på 10 bar till en första syrgasmixer(+ ånga) följt av efterbehandling i en "pre retention tube" (förreaktor) med 10 minuters uppehållstid där massatrycket reduceras till en nivå runt 8-6 bar p.g.a. rörförluster etc. Därefter matades massan till en andra syrgasmixer följt av efterbehandling i en reaktor vid trycket 5-2 bar samt uppehållstid 60 minuter. Här angavs att man helst hade velat ha en "pre retention tube" som skulle gett en uppehållstid om 20 minuter, men att det inte kunnat utföras på grund av utrymmesbrist. OJI PAPER angav att de genom denna installation lyckats med ökad kappareduktion till minskad kemikaliekostnad samt förbättrad massaviskositet.

Huvuddelen av den kända tekniken har således varit inriktad på ett högre tryck i den första reaktorn på en nivå runt 6(8)-10 bar. I vissa extrema applikationer har till och med ett tryck i den första reaktorn på upp till 20 bar diskuterats. Detta medför att de reaktorutrymmen som erfordras för den första delignifieringszonen måste tillverkas för att klara av dessa höga trycknivåer med åtföljande behov av kraftig godstjocklek och/eller goda materialkvalitéer, vilket medför en dyr installation.

I massasuspensioner vid industriella framställningsprocesser finns det stora mängder med lätt oxiderbara beståndsdelar/strukturer vilka reagerar redan vid modesta processbetingelser. Det är därför fördelaktigt att man i ett första steg satsar syrgas i

sådana mängder att denna relativt lättoxiderade del av massan tillåtes oxidera/reagera först. Stora problem uppstår om man försöker kompensera detta genom översatsning av syrgas, då kanaliseringsproblematiken (såsom anges i den nämnda SE,C,505.147) är överhängande.

Ändamålet med uppfinningen

Ett ändamål med uppfinningen är att undvika nackdelarna med den kända tekniken samt att erhålla en syrgasdelignifiering med ökad selektivitet. Uppfinningen medger en optimal praktisk tillämpning av teorierna om en första snabb fas och en andra långsammare fas under syrgasdelignifieringsprocessen, där de optimala reaktionsbetingelserna är skiljaktiga mellan faserna.
Vid de konventionellt tillämpade höga hydroxidjonkoncentrationerna och höga syrgaspartialtrycken i det första steget attackeras kolhydraterna mer än nödvändigt vilket försämrar massakvaliteten. Ett lägre syrgaspartialtryck, och företrädesvis även lägre temperatur, i det första steget än i det andra steget minskar reaktionshastigheten för nedbrytning av kolhydrater mer än det minskar reaktionshastigheten för delignifieringen, vilket leder till ökad total selektivitet på massan efter de två stegen.

Ytterligare ett ändamål är att medge en enklare och billigare processinstallation, där åtminstone ett tryckkärl i en första delignifieringszon kan tillverkas med klenare gods och/eller med lägre materialkvalitet anpassat för en lägre tryckklass.

Ännu ett ändamål är att möjliggöra användning även av ånga med måttligt tryck, speciellt när man behöver höja temperaturen mycket mellan första och andra steget, och där det andra steget håller ett relativt första steget betydligt högre tryck. Tillgången på medltrycksånga och lågtrycksånga är i de flesta fall mycket god på massabruken, emedan högtrycksånga är en bristvara på grund av de flertal processer som kräver högtrycksånga. Detta möjliggör även ombyggnationer av befintliga enkärls delignifieringssystem där man med tidigare känd teknik för ombyggnad till tvåstegsutförande varit begränsad av att det rådande trycket på anläggningens ångnät inte medgivit att tillräckligt stor mängd ånga kunnat blandas in i massan för att nå

önskvärd temperatur i det andra delignifieringssteget.

Ännu ett ändamål är att optimera mixningsprocessen i respektive position så att endast den mängd kemikalie/syrgas tillsättes, vilken åtgår i den efterföljande delignifieringszonen, och där inmixningen av kemikalier/syrgas ej behöver konkurrera med samtidig inmixning av ånga i syfte att höja temperaturen till önskad nivå. Härigenom kan man undvara blödningssystem för överskjutande mängder av syrgas samtidigt som man kan reducera den totala åtgången av syrgas, vilket reducerar driftkostnaderna för operatören av fiberlinjen och sålunda kortar ner pay-off tiden. Samtidigt kan man välja en mindre storlek på en dynamisk mixer för inmixning av kemikalier, vilken enbart dimensioneras för aktuella inmixningsvolymer av kemikalier.

Ännu ett ändamål är att i ett syrgasdelignifieringssystem med viss sammanlagd volym av det första och andra steget, höja en så kallad H-faktor, genom att det första steget körs en kort tid vid låg temperatur och det andra steget vid en längre tid vid högre temperatur. Vid exempelvis ombyggnationer av befintliga enkärls syrgasdelignifieringssteg så kan en enkel ombyggnad med en liten förreaktor och modest höjning av reaktionstemperaturen i den befintliga reaktorn höja H-faktorn och samtidigt förbättras selektiviteten över syrgasstegen.

Uppfinningen beskrives närmare med hänvisning till figurer enligt följande figurförteckning.

<u>Figurförteckning</u>

Figur 1; visar ett system för syrgasdelignifiering i två steg enligt uppfinningen; OCH
Figur 2; visar schematiskt syrgasdelignifieringens kinetik samt vilka fördelar som erhålles relativt känd teknik vad avser kappatalsreduktion samt höjd H-faktor.

<u>Beskrivning av utföringsexempel</u>

I figur 1 visas en uppfinningsenlig installation av ett system i en befintlig anläggning där syrgasdelignifieringsprocessen krävde en uppgradering.

En befintlig första MC-pump 1 (MC=Medium Consistency, typiskt 8-18% massakonsistens) är ansluten till ett fallrör 2 för vidare befordran till en befintlig första MC-mixer 3. Den första mixern 3 är en så kallad dynamisk mixer, där en motordriven rotor agiterar massan i åtminstone en trång fluidiseringsspalt. Den dynamiska mixern är företrädesvis en mixertyp motsvarande den som visas i US433920, där en första cylindrisk fludiseringszon bildas mellan rotor och hus och en andra fludiseringszon bildas mellan en radiellt riktad rotordel och huset, vilken mixer härmed är införd som referens. En mekanisk agitation erfodras för att få en jämn inblandning av aktuell kemikaliesatsning i hela massasuspensionen, i syfte att massan skall blekas/behandlas jämt genom hela massavolymen.

I den första MC-mixern 3 sker en inblandning av kemikalier, främst syrgas, varefter i det befintliga systemet massan matades till en syrgasreaktor 6.

Kombinationen av en första MC-pump 1 tätt följt av en MC-mixer 3 kan benämnas ”perfect pair”. Detta då pumpen primärt ger en viss trycksättning av massflödet vilket underlättar en finfördelad tillförsel av syrgasen i den direkt därefter följande MC-mixern.

I enlighet med uppfinningen erhålles en uppgradering av syrgasdelignifieringsprocessen genom att införa en statisk mixer 8, dvs en icke roterande eller mekaniskt agiterande mixer 8 för temperaturhöjning genom tillsats av ånga. Den statiska mixern 8 är företrädevis av en konstruktion som visats i SE,C,512.192(=PCT/SE00/00137), där ånga leds in som tunna strålar/"jets" genom ett flertal hål jämt fördelade över omkretsen på ett massabefodrande rör, vilken mixer härmed är införd som referens.

Den statiska mixern 8 är anordnad direkt efter syrgasreaktorn 6, och följt av en andra MC-pump 4 samt en omedelbart därefter verkande andra agiterande MC-mixer 5 av samma typ som mixern 3. Systemet sammanbygges så att sammankopplingsröret 6 bildar en första delignifieringszon mellan den första MC-mixerns 3 utlopp och den icke roterande mixerns 8 inlopp vilken zon medför en uppehållstid R_T om 2-20 minuter, företrädesvis 2-10 minuter och än mer fördelaktigt 3-6 minuter.

Den andra MC-pumpen 4 regleras så att det resulterande trycket i uppehållsledningen 6 företrädesvis ligger i intervallet 0-6 bar, företrädesvis 0-4 bar. Företrädevis regleras den

andra pumpen 4 genom en varvtalsreglering av ett reglersystem PC i beroende av i den första delignifieringszonen 6 rådande och detekterade trycket.
Temperaturen i hela den första delignifieringszonen 6 kan hållas låg, företrädesvis på den nivå som systemet medger utan tillsättning av ånga, dock företrädesvis en ingående temperatur på massan till den första delignifieringszonen runt 85 °C, +/- 10 °C.

Efter den första delignifieringszonen ansluter den icke roterande mixern 8 samt den andra MC-pumpen 4 följt av den andra MC-mixern 5. Denna andra "perfect pair"-kombination regleras så att det resulterande trycket i syrgasreaktorn 10, vilken bildar en andra delignifieringszon, når en nivå om minst 3 bars övertryck i toppen på reaktorn. Trycket i den andra MC-mixern skall vid konventionell applikation vara minst 4 bar högre relativt trycket i den första MC-mixern, alternativt att tryckhöjningen i den andra pumpen når 4 bar. Vid praktisk tillämpning i konventionella syrgassteg erhåller ett initialtryck inom intervallet 8-10 bar, motsvarande trycket vid reaktorns inlopp.

I enlighet med uppfinningen ökas temperaturen på massan i den andra delignifieringszonen genom att tillföra ånga direkt efter den första delignifieringszonen i den icke roterande mixern, och innan den tryckhöjande pumpen 4 tar vid. Ångtillförseln regleras lämpligen med ett reglersystem TC innefattande en reglerventil V på ledningen 7 för ångtillförseln samt en återkopplande temperaturmätning på den från mixern utgående massan. Temperaturen höjs lämpligen till en nivå om 100 °C +/- 10 °C, dock företrädesvis minst 5 °C högre än temperaturen i den första delignifieringszonen. Genom att ångtillförseln utföres innan massan ges det högre trycket vilket är nödvändigt för slutfasen av delignifieringen:
- kan högre temperatur erhållas
- krävs lägre tryck på tillgänglig ånga
- behöver ej mixrarna för kemikaliesatsningen/syrgasinblandningen belastas med tillförsel även av ånga, vilket reducerar blandarnas effektivitet.

Volymen på den andra delignifieringszonen, d.v.s. andra reaktorn, utformas lämpligen så att den är åtminstone 10 gånger större än volymen i den första delignifieringszonen,

d.v.s. minst 20-200 minuter, företrädesvis 20-100 minuter och än mer fördelaktigt i området 50-90 minuter.

I figur 2 visas schematiskt syrgasdelignifieringens kinetik samt vilka fördelar som erhålles relativt känd teknik vad avser principerna för kappatalsreduktion.
Med kurva P1 redovisas ett principiellt reaktionsförlopp under delignifieringens initialfas. Denna del av delignifieringen går relativt snabbt och har väsentligen fullbordats typiskt efter drygt 20 minuter.
Efter en kortare tid, typiskt bara 5-10 minuter, tar dock delignifieringens slutfas P2 över och börjar dominera vad avser den på massan resulterande delignifieringen.
Vid strecket A visas en typisk indelning av delignifieringen i två steg enligt känd teknik, med steg 1 till vänster om strecket A samt steg 2 till höger om strecket A. Härav framgår att i steg 1 sker egentligen två olika dominerande processer, dels delignifieringens initialfas men även dess slutfas. Av detta kan man dra slutsatsen att det blir omöjligt att optimera processbetingelserna i steg 1 för båda dessa delignifieringsfaser.
Vid strecket B visas istället en indelning av delignifieringen i två steg i enlighet med uppfinningen, med steg 1 till vänster om strecket B och steg 2 till höger om strecket B. Härigenom kan man optimera respektive steg för den i steget dominerande processen.
Kurvan H_A visar typiskt den temperaturintegral med avseende på tid (H-faktor) som erhålles vid tillämpning av en delignifieringsprocess i två steg enligt känd teknik motsvarande strecket A.
Som framgår av figuren kan man erhålla en relativt sett högre H-faktor med den uppfinningsenliga stegindelningen jämfört med den i dagens installationer typiska. Detta kan göras utan avkall på krav på hög selektivitet över syrgasdelignifierings-systemet.
Uppfinningen öppnar även vägar för att med en liten investering uppgradera ett befintligt relativt sett lågselektivt 1-stegsförfarande till ett 2-stegssystem med bättre selektivitet, detta utan att behöva bygga en eller t.o.m. två nya stora reaktorer. Enligt uppfinningen klaras initialfasen av syrgasdelignifieringen av i förreaktorn varefter temperaturen i den befintliga reaktorn om så behövs t.o.m. kan höjas vid ombyggnad

och en förhöjd H-faktor på detta vis kan kombineras med förhöjd selektivitet.

Uppfinningen kan modifieras på ett flertal sätt inom ramen för uppfinningstanken. Exempelvis så kan den första delignifieringszonen bestå av en vertikal ”pre retention tube”, men där trycket i någon del av denna "pre retention tube", även i dess botten, är minst 4 bar lägre än trycket i den andra delignifieringszonens initialdel.
Ytterligare delignifieringszoner eller mellanliggande tvättning/lakning eller extraktion av massan kan införas mellan den uppfinningsenliga första och andra delignifieringszonen. Exempelvis kan en tredje ”perfect pair”-kombination, d.v.s. pump med efterföljande mixer, anordnas mellan zonerna. Det väsentliga är att den första delignifieringszonen ges ett lägre tryck, kort uppehållstid samt måttlig temperatur, och där den avslutande sista delignifieringszonen ges ett högre tryck (minst 4 bar högre tryck än första zonen), längre uppehållstid (minst 10 gånger längre tid än första zonen) samt förhöjd temperatur (företrädesvis minst 5 grader högre temperatur än den första zonen).
Eventuellt skulle en första mixer eller en mellanliggande mixer i en tredje ”perfect pair”-kombination kunna satsas med syrgas som, åtminstone till viss del, blåses av från reaktorn 10. De ekonomiska förutsättningarna för en sådan återvinning av syrgas är dåliga, då kostnaden för syrgas är relativt låg.

För att säkerställa optimala processbetingelser så kan endera, företrädesvis den andra eller båda av MC-pumparna vara varvtalsreglerade i beroende av trycket i den första delignifieringszonen.

Uppfinningen kan även modifieras med ett antal olika tillkommande tillsättningar av andra kemikalier tillsammans med syrgasen, eller skilt från syrgastillsatsen i en separat tillsättningsposition, vilka kemikalier är valda och anpassade till den specifika fiberlinjen och aktuell massakvalitet, såsom
-alkali/NaOH för justering av pH-nivån till det som är lämpligt för aktuell massakvalitet,
-skyddsmedel för cellulosa, exempelvis $MgSO_4$, eller andra alkaliska jordartsmetall-

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joner eller föreningar därav;

-komplexbildartillsatser före syrgastillsättning med en eventuell åtföljande avskiljning av utfällda metaller;

-klordioxid;

-väteperoxid eller organiska eller oorganiska persyror eller salter därav;

-radikalfångare, såsom alkoholer, ketoner, aldehyder eller organiska syror; samt

-koldioxid eller andra tillsatser.

Eventuellt skulle även en avluftning av avgaser(restgaser) kunna ske i omedelbar anslutning till den andra pumpen, företrädesvis genom att pumpen är försedd med intern avluftning, företrädesvis en pump benämnd "degassing pump".

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PATENTKRAV

1. System för syrgasdelignifiering av massa av lignocellulosahaltigt material vid medelkoncentration 8-18% på massan , vilken syrgasdelignifiering sker i åtminstone två steg och där systemet kännetecknas av att det innefattar;
- en första pump (1) anordnad att pumpa massan till en första mixer (3) för inblandning av för syrgasdelignifieringen nödvändiga kemikalier i denna första mixer (3), vilken första mixer är anordnad i nära anslutning till den första pumpen,
- en första delignifieringszon (6) anordnad att mottaga massa från den första mixern (3),
- en andra mixer (8) anordnad att mottaga massa från den första delignifieringszonen, vilken andra mixer innefattar medel för inblandning av ånga (MP-Steam) till massan ,
- en andra pump (4) anordnad att mottaga massa efter den andra mixern,
- en tredje mixer (5) anordnad i nära anslutning till den andra pumpen, för inblandning av för syrgasdelignifieringen nödvändiga kemikalier i denna tredje mixer (5)
- en andra delignifieringszon (10) anordnad att mottaga massan från den tredje mixern.

2. System för syrgasdelignifiering enligt krav 1 kännetecknat av att den första och tredje mixern (3 respektive 5) är en mixer med mekanisk agitation, där massan åtminstone delvis fluidiseras i spalter i mixern, samt att den andra mixern (8) är en statisk mixer utan mekanisk agitation.

3. System för syrgasdelignifiering enligt krav 2 kännetecknat av att det innefattar medel (O_2) för satsning av syrgas i den första (3) respektive tredje mixern (5) samt medel (MP-Steam) för satsning av ånga i den andra mixern (8).

4. System för syrgasdelignifiering enligt krav 3 kännetecknat av att den andra mixern innefattar medel för reglerbar ångtillförsel (7,V,TC) företrädesvis återkopplat styrt i beroende av temperaturen på massan efter sagda mixer.

5. System för syrgasdelignifiering enligt krav 4 kännetecknat av att den

andra mixern (8) utgöres av ett massabefordrande rör med flera inloppshål för ångan i rörets vägg.

6. System för syrgasdelignifiering enligt krav 5 kännetecknat av att ångan utgöres av mellantrycksånga vid 8-14 bar.

7. System för syrgasdelignifiering enligt krav 3 kännetecknat av att systemet innefattar ett reglersystem (PC) för reglering av varvtalet på den andra pumpen (4) beroende av trycket i den första delignifieringszonen (6).

8. System för syrgasdelignifiering enligt krav 1 kännetecknat av
- att den första delignifieringszonen (6) har en volym som medför en uppehållstid om 2-20 minuter, företrädesvis 2-10 minuter och än mer fördelaktigt 3-6 minuter för massan i den första delignifieringszonen,
- att systemet är så avpassat på så sätt att trycket i den första delignifieringszonen uppgår till 0-6 bar, företrädesvis 0-4 bar,
- att den andra pumpen (4) har en pumpeffekt så att trycket i den andra delignifieringszonen, når en nivå om minst 3 bars övertryck i toppen på i den andra delignifieringszonen, och
- att den andra delignifieringszonen (10) har en volym som är minst 10 gånger större än volymen i den första delignifieringszonen, d.v.s. har en volym som medför en uppehållstid om minst 20-200 minuter, företrädesvis 20-100 minuter och än mer fördelaktigt i området 50-90 minuter.

9. Förfarande för syrgasdelignifiering av massa av lignocellulosahaltigt material vid medelkoncentration 8-18% på massan i åtminstone två steg
kännetecknat av
a) att massa vid medelkoncentration trycksättes och
b) därefter tillsättes kemikalier för syrgasdelignifieringen, främst syrgas, så att syrgasdelignifiering sker i ett första steg där massan behandlas under en kortare tid motsvarande 2-20 minuter, företrädesvis 2-10 minuter och än mer fördelaktigt 3-6

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minuter, under måttligt övertryck inom intervallet 0-6 bar, företrädesvis 0-4 bar, samt måttlig temperatur i området 85 °C +/- 10 °C,

c) och att massan efter det första steget och vid medelkoncentration på massan först sammanblandas med ånga i syfte att höja temperaturen,

d) varefter följer en först en trycksättning av den uppvärmda massan

e) och därefter en andra tillsättning av kemikalier för syrgasdelignifieringen, främst syrgas,

f) för att i ett avslutande steg behandlas under en relativt första steget längre tid, i storleksordningen 10 gånger längre tid än första steget, i intervallet 20-200 minuter, företrädesvis 20-100 minuter och än mer fördelaktigt i intervallet 50-90 minuter samt sker vid ett initialtryck inom intervallet 8-10 bar, motsvarande trycket vid reaktorns inlopp men även högre temperatur, företrädesvis i området 100 °C +/- 10 °C, dock företrädesvis minst 5 °C högre än temperaturen i det första steget.

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SAMMANDRAG

Uppfinningen avser ett system samt förfarande för syrgasdelignifiering av massa av lignocellulosahaltigt material vid medelkoncentration på massan vid 8-18% massakonsistens, i åtminstone två steg.

Uppfinningen utmärkes av att syrgasdelignifiering sker i en processekvens bestående av:

- en första pump (1), följt av
- en första syrgasmixer (3), följt av
- en första delignifieringszon (6)

och direkt därefter följt av

- en andra ångmixer (8), följt av
- en andra pump (4), följt av
- en tredje syrgasmixer (5) och slutligen
- en andra delignifieringszon (10).

Härigenom kan man i en industriell process utnyttja syrgasdelignifieringens kinetik på ett optimalt sätt för erhållande av en selektiv syrgasdelignifiering, till låg installationskostnad och låg driftskostnad, även om endast lågtrycksånga finns tillgänglig.

(Fig.1)

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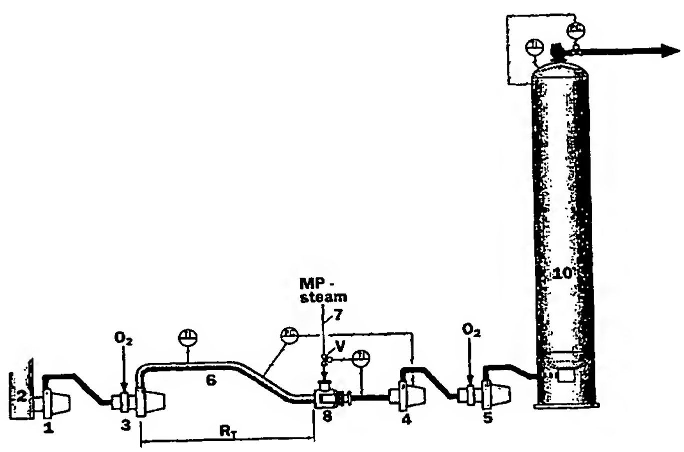
(54) Title: SYSTEM AND PROCESS FOR THE OXYGEN DELIGNIFICATION OF PULP CONSISTING OF LIGNOCELLULOSE-CONTAINING MATERIAL



(57) Abstract: The invention relates to a system and a process for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18 % pulp consistency, in at least two stages. The invention is characterized in that oxygen delignification takes place in a process sequence consisting of: a first lamp (1), followed by a first oxygen mixer (3), followed by a first delignification zone (6) and, directly afthat, followed by a second steam mixer (8), followed by a second pump (4), followed by a third oxygen mixer (5) and, finally, a second delignification zone (10). This makes it possible, in an industrial process, to exploit the kinetics of oxygen delignification optimally in order to obtain selective oxygen delignification at low installation cost and low operating cost, even if only low-pressure steam is available.

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A. CLASSIFICATION OF SUBJECT MATTER

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 9715715 A1 (SUNDS DEFIBRATOR INDUSTRIES AB), 1 May 1997 (01.05.97), page 1, line 33 - page 3, line 12, figure 1	1-8
A		9
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Y	WO 9630586 A1 (KVAERNER PULPING AB), 3 October 1996 (03.10.96), page 2, line 25 - line 32; page 3, line 29 - page 5, line 21	1-8
A		9
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A	SE 8006701 A (SCA DEVELOMENT AKTIEBOLAG), 26 March 1982 (26.03.82)	1-9
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☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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				CN	1200154	A	25/11/98
				EP	0857231	A	12/08/98
				JP	11514410	T	07/12/99
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				NZ	320687	A	25/11/98
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SE	8006701	A	26/03/82	NONE			

SYSTEM AND PROCESS FOR THE OXYGEN DELIGNIFICATION OF PULP CONSISTING OF LIGNOCELLULOSE-CONTAINING MATERIAL

The present invention relates to a system and a process for oxygen delignification in accordance with the preambles to patent claim 1 and patent claim 9, respectively.

State of the art

A number of different processes for oxygen delignification are known.

US,A,4.259.150 presents a system with multistage oxygen bleaching in which, in each stage, the pulp is first mixed to a lower consistency with O_2, water and NaOH, followed by a thickening back to the consistency level which the pulp had prior to the stage in question. The aim is to obtain an economic, chlorine-free bleaching with high yield. At the same time, the kappa number can be lowered, by means of repeated stages, from 70 down to 15 or even less than 15.

SE,C,467.582 presents an improved system for the oxygen bleaching of pulp of medium consistency. By means of controlling the temperature in an optimized manner, an oxygen bleaching takes place in a first delignification zone at a low temperature, with this being followed by a second delignification zone at a temperature which is 20-40 degrees higher. The aim is to obtain an improved yield and an improved viscosity, while retaining the dwell time, in association with industrial use.

Other variants of oxygen delignification in two stages have also been patented in addition to SE,C,467.582. SE,C,505.147 presents a process in which the pulp should have a high pulp concentration in the range of 25-40% in the first stage and a concentration of 8-16% in the second stage, at the same time as the

temperature in the second stage should be higher than, or equal to, the temperature in the first stage, in line with the temperature difference which is recommended in SE,C,467.582. The advantages of the solution in accordance with SE,C,505.147 are stated to be the possibilities of admixing more oxygen in the first high-consistency stage without there being any risk of channel formation but where, at the same time, unused quantities of oxygen can be bled off after the first stage in order subsequently to be admixed in a second mixer prior to the second stage.

SE,C,505.141 presents a further process which is an attempt to circumvent SE,C,467.582, since that which it is sought to patent is stated to be that a temperature difference between the stages does not exceed 20 degrees, i.e. the lower suitable temperature difference patented in SE,C,467.582, but that a temperature difference should nevertheless be present. In addition to that, it is stated that a) the pressure should be higher in the first stage and b) that the dwell time is short in the first stage, i.e. in the order of magnitude of 10-30 minutes, and also c) the dwell time in the second stage is longer, i.e. in the order of magnitude of 45-180 minutes.

A lecture entitled "*Two stage MC-oxygen delignification process and operating experience*", which was given by Shinichiro Kondo from the Technical Div. Technical Dept. OJI PAPER CO. Ltd. At the *1992 Pan-Pacific Pulp & Paper Technology Conference ('99 PAN-PAC PPTC), Sept. 8-10, Sheraton Grande Tokyo Bay Hotel & Towers,* presents a successful installation which was constructed with two-stage oxygen delignification in 1986 in a plant in Tomakomai.

In this OJI PAPER plant in Tomakomai, the pulp was fed, with a pressure of 10 bar, to a first oxygen mixer (+ team) followed by an after-treatment in a "preretention tube" (prereactor), with a 10 minute

dwell time in which the pulp pressure is reduced to a level of about 8-6 bar due to pipe losses, etc. After that, the pulp was fed to a second oxygen mixture followed by an after-treatment in a reactor at a pressure of 5-2 bar and with a dwell time of 60 minutes. It was stated at this point that preference would have been given to having a "preretention tube" which would have given a dwell time of 20 minutes but that it was not possible to construct this due to lack of space. OJI PAPER stated that, by using this installation, they had succeeded in obtaining an increase, in kappa reduction at a lower cost in chemicals and with the pulp viscosity being improved.

Most of the the prior art has consequently been directed towards a higher pressure in the first reactor at a level of about 6(8)-10 bar. A pressure in the first reactor of up to 20 bar has even been discussed in certain extreme applications. This results in it being necessary to manufacture the reactor spaces which are required for the first delignification zone such that they can cope with these high pressure levels, with a consequent requirement for substantial material thickness and/or good material qualities, which in turn result in an expensive installation.

In pulp suspensions in industrial production processes, there are large quantities of readily oxidizable constituents/structures which already react under modest process conditions. It is therefore advantageous, in a first stage, to add oxygen in quantities which are such that this part of the pulp which is relatively easily oxidized is allowed to oxidize/react first of all. Severe problems arise if an attempt is made to compensate for this by overadding oxygen since there is the immediate danger of canalization problems (as mentioned in the said SE,C,505.147).

Object of the invention

One object of the invention is to avoid the disadvantages of the prior art and to obtain an oxygen delignification which gives increased selectivity. The invention permits an optical practical application of the theories regarding a first rapid phase and a second slower phase during the oxygen delignification process, with the optimal reaction conditions being different between the phases.

At the high hydroxide ion concentrations and high oxygen partial pressures which are conventionally employed in the first stage, the carbohydrates are attacked more than is necessary, thereby impairing the quality of the pulp. A lower oxygen partial pressure, and preferably a lower temperature as well, in the first stage as compared with the second stage decreases the rate of reaction for the breakdown of carbohydrates more than it decreases the rate of reaction for the delignification, leading in turn to an increase in the total selectivity on the pulp after the two stages.

Another object is to allow a simpler and cheaper process installation in which at least one pressure vessel, in a first delignification zone, can be manufactured using thinner material and/or using a lower material quality which is suitable for a lower pressure class.

Yet another object is also to make it possible to use steam at moderate pressure especially when there is a need to increase the temperature substantially between the first and second stage and when the pressure in the second stage is considerably higher than that in the first stage. In most cases, the supply of medium-pressure steam and low-pressure steam is very good in a pulp mill whereas high-pressure steam is in short supply due to the large number of processes which require high-pressure steam. This also makes it

possible to convert existing single-vessel delignification systems where, with the previously the prior art for converting to a two-stage design, a restriction has been imposed by the fact that the prevailing pressure in the plant's steam grid has not enabled a sufficiently large quantity of steam to be admixed with the pulp in order to achieve the desired temperature in the second delignification stage.

Yet another object is to optimize the mixing process in each position such that only that quantity of chemicals/oxygen is added which is consumed in the subsequent delignification zone and where the admixture of chemicals/oxygen does not need to compete with the simultaneous admixture of steam for the purpose of increasing the temperature to the desired level. In this way, it is possible to dispense with bleeding systems for overshooting quantities of oxygen at the same time as it is possible to reduce the total consumption of oxygen, which in turn reduces the operating costs for the operator of the fibre line and thus shortens the pay-off time. At the same time it is possible to select a smaller size of dynamic mixer for admixing chemicals, which mixer is dimensioned solely for the volumes of chemicals which are actually being admixed.

Yet another object is to increase, in an oxygen delignification system having a certain total volume of the first and second stages, a so-called H factor by operating the first stage for a short time at low temperature and operating the second stage for a longer time at a higher temperature. Thus, in connection, for example, with conversions of existing single-vessel oxygen delignification stages, a simple conversion, including a small prereactor and a modest increase in the reaction temperature in the existing reactor, can increase the H factor and at the same time improve the selectivity over the oxygen stages.

The invention is described in more detail with reference to the figures in accordance with the following figure list.

Figure list

Figure 1 shows a system for oxygen delignification in two stages in accordance with the invention; AND

Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages which are gained relative to the prior art with regard to reduction in kappa number and an increased H factor.

Description of embodiment examples

Figure 1 shows an installation, according to the invention, of a system in an existing plant in which the oxygen delignification process needed upgrading.

An existing first MC pump 1 (MC = medium consistency, typically a pulp consistency of 8-18%) is connected to a tipping chute 2 for forwarding to an existing first MC mixer 3. The first mixer 3 is a so-called dynamic mixer, in which a motor-driven rotor agitates the pulp in at least one narrow fluidization gap. The dynamic mixer is preferably a mixer type which corresponds to that which is shown in US433920, in which a first cylindrical fluidization zone is formed between the rotor and the housing and a second fluidization zone is formed between a radially directed rotor part and housing, which mixer is hereby introduced as a reference. A mechanical agitation is required in order to obtain a uniform admixture of the chemical charge in question in the whole of the pulp suspension, with the aim of the pulp being bleached/treated uniformly throughout the whole of the volume of the pulp.

An admixture of chemicals, chiefly oxygen, takes place in the first MC mixer 3, after which the pulp was, in the existing system, fed to an oxygen reactor 6.

The combination of a first MC pump 1 followed closely by an MC mixer 3 can be termed a "perfect pair". This is the case since the pump primarily pressurizes the pulp flow to a given degree, thereby facilitating a finely divided supply of the oxygen to the MC mixer which follows directly thereafter.

In accordance with the invention, an upgrading of the oxygen delignification process is achieved by introducing a static mixer 8, i.e. a non-rotating or mechanically agitating mixer 8 for increasing the temperature by means of adding steam. The static mixer 8 is preferably of a construction which has been shown in SE,C,512.192 (= PCT/SE00/00137), where steam is conducted in as thin jets through a number of holes which are uniformly distributed over the periphery of a pulp-conveying pipe, which mixer is hereby introduced as a reference.

The static mixer 8 is arranged directly after the oxygen reactor 6 and followed by a second MC pump 4 and a second agitating MC mixer 5, of the same type as the mixer 3, which acts directly after the MC pump 4. The system is assembled such that the coupling pipe 6 forms a first delignification zone between the outlet of the first MC mixer 3 and the inlet of the non-rotating mixer 8, which zone gives rise to a dwell time R_T of 2-20 minutes, preferably 2-10 minutes and even more advantageously 3-6 minutes.

The second MC pump 4 is controlled such that the resulting pressure in the dwell line 6 is preferably in the interval 0-6 bar, preferably 0-4 bar. Preferably, the second pump 4 is controlled by means of its rotational speed being controlled by a control system PC depending on the pressure which prevails, and is detected, in the first delignification zone 6.

The temperature in the whole of the first delignification zone 6 can be kept low, preferably at the level which the system allows without adding steam, but preferably with the pulp entering the first delignification zone being at a temperature of about 85°C, ± 10°C.

The non-rotating mixer 8 is connected in after the first delignification zone, as are then the second MC pump 4 followed by the second MC mixer 5. This second "perfect pair" combination is controlled such that the resulting pressure in the oxygen reactor 10, which forms a second delignification zone, reaches a level of at least 3 bars overpressure at the top of the reactor. In conventional applications, the pressure in the second MC mixer should be at least 4 bar higher than the pressure in the first MC mixer; alternatively, the increase in pressure in the second pump should reach 4 bar. In connection with practical implementation in conventional oxygen stages, an initial pressure is obtained within the interval 8-10 bar, corresponding to the pressure at the inlet to the reactor.

In accordance with the invention, the temperature of the pulp in the second delignification zone is increased by supplying steam to the non-rotating mixer directly after the first delignification zone and before the pressure-raising pump 4 comes into play. The steam supply is expediently controlled using a control system TC, which comprises a control valve V on the line 7 for the steam supply and a feeding-back measurement of the temperature of the pulp which is leaving the mixer. The temperature is expediently raised to a level of 100°C ± 10°C, but preferably at least 5°C higher than the temperature in the first delignification zone. As a result of the steam being added before the pulp is given the higher pressure

which is required for the final phase of the delignification:

- a higher temperature can be obtained
- the pressure of the available steam does not need to be so high
- the mixers for adding chemicals/admixing oxygen do not need to be burdened with a supply of steam as well, which will otherwise reduce their efficiency.

The volume of the second delignification zone, i.e. the second reactor, is expediently designed such that it is at least 10 times greater than the volume of the first delignification zone, i.e. a retention time of at least 20-200 minutes, preferably 20-100 minutes and even more advantageously within the range 50-90 minutes.

Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages with regard to the principles of kappa number reduction which are obtained relative to the prior art. Curve P1 shows the principle of a reaction course during the initial phase of the delignification. This part of the delignification proceeds relatively rapidly and is typically essentially complete after a good 20 minutes. However, after a relatively short time, typically only 5-10 minutes, the final phase P2 of the delignification takes over and begins to dominate as far as the resulting delignification of the pulp is concerned. A typical subdivision of the delignification into two stages in accordance with the prior art is shown at line A, with stage 1 being to the left of the line A and stage 2 being to the right of the line A. It follows from this that two different dominating processes, i.e. the initial phase of the delignification on the one hand, but also its final phase, actually take place in stage 1. It can be concluded from this that it becomes impossible to

optimize the process conditions in stage 1 for both these delignification phases.

Instead, a subdivision of the delignification into two stages in accordance with the invention is shown as a line B, a stage 1 is to the left of the line B and stage 2 is to the right of the line B. This makes it possible to optimize each stage for the process which dominates in the stage. The curve H_A shows the temperature integral plotted against time (the H factor) which is typically obtained when implementing a delignification process in two stages in accordance with the prior art, corresponding to the line A.

As can be seen from the figure, it is possible to use the stage subdivision in accordance with the invention to obtain an H factor which is higher than that which is typically obtained in current installations. This can be done without foregoing demands for high selectivity over the oxygen delignification system.

The invention also opens up ways of upgrading, with a small investment, an existing 1-stage process of comparatively low selectivity to a 2-stage system of better selectivity without having to build a new large reactor or even two such reactors. According to the invention, the initial phase of the oxygen delignification is dealt with in the prereactor, after which the temperature in the existing reactor can even be increased, if so required, in association with the conversion, and an increased H factor can in this way be combined with increased selectivity.

The invention can be modified in a number of ways within the context of the inventive concept. For example, the first delignification zone can consist of a "preretention tube" which is vertical but in which the pressure in some part of this "preretention tube", including its bottom, is at least 4 bar lower than the pressure in the initial part of the second delignification zone.

Further delignification zones, or intermediate washing/bleaching or extraction of the pulp, can be introduced between the first and second delignification zones according to the invention. For example, a third "perfect pair" combination, i.e. a pump with a mixer following it, can be arranged between the zones. What is essential is that the first delignification zone is characterized by a lower pressure, a short dwell time and a moderate temperature, and that the concluding, final delignification zone is characterized by a higher pressure (a pressure which is at least 4 bar higher than that of the first zone), a longer dwell time (a dwell time which is at least 10 times longer than that in the first zone) and an increased temperature (a temperature which is preferably at least 5 degrees higher than that in the first zone).

Where appropriate, it should be possible to charge a first mixer, or an intermediate mixer in a third "perfect pair" combination, with oxygen, at least some part of which is blown off from the reactor 10. The economic basis for such a recovery of oxygen is poor since the cost of oxygen is relatively low.

In order to ensure optimal process conditions, one or other, preferably the second, or both of the MC pumps can be rotation speed-controlled in dependence on the pressure in the first delignification zone.

The invention can also be modified by a number of varying additions of other chemicals either together with the oxygen or separately from the addition of oxygen, in a separate adding position, which chemicals are selected and suitable for the specific fibre line and the pulp quality in question, such as

- alkali/NaOH for adjusting the pH level to that which is suitable for the pulp quality in question,
- agents for protecting cellulose, for example $MgSO_4$ or other alkaline earth metal ions or compounds thereof;

- additions of complexing agents which are performed prior to adding oxygen, with subsequent removal of precipitated metals, where appropriate,
- chlorine dioxide;
- hydrogen peroxide or organic or inorganic peracids or salts thereof;
- free-radical capturing agents, such as alcohols, ketones, aldehydes or organic acids; and
- carbon dioxide or other additives.

Where appropriate, it should also be possible to degas exhaust gases (residual gases) in immediate conjunction with the second pump, preferably by means of the pump being provided with internal degassing, preferably a pump termed a "degassing pump".

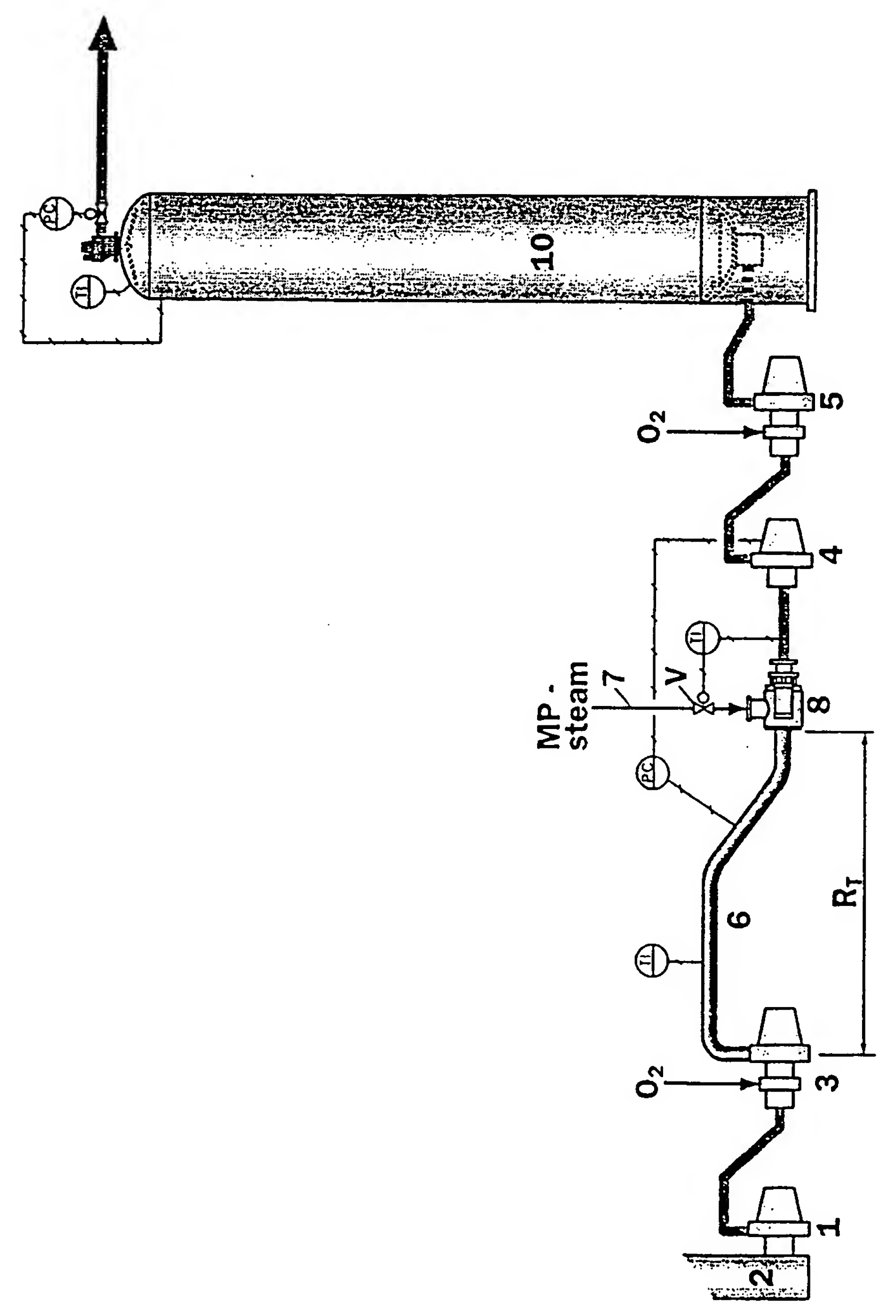

Fig1

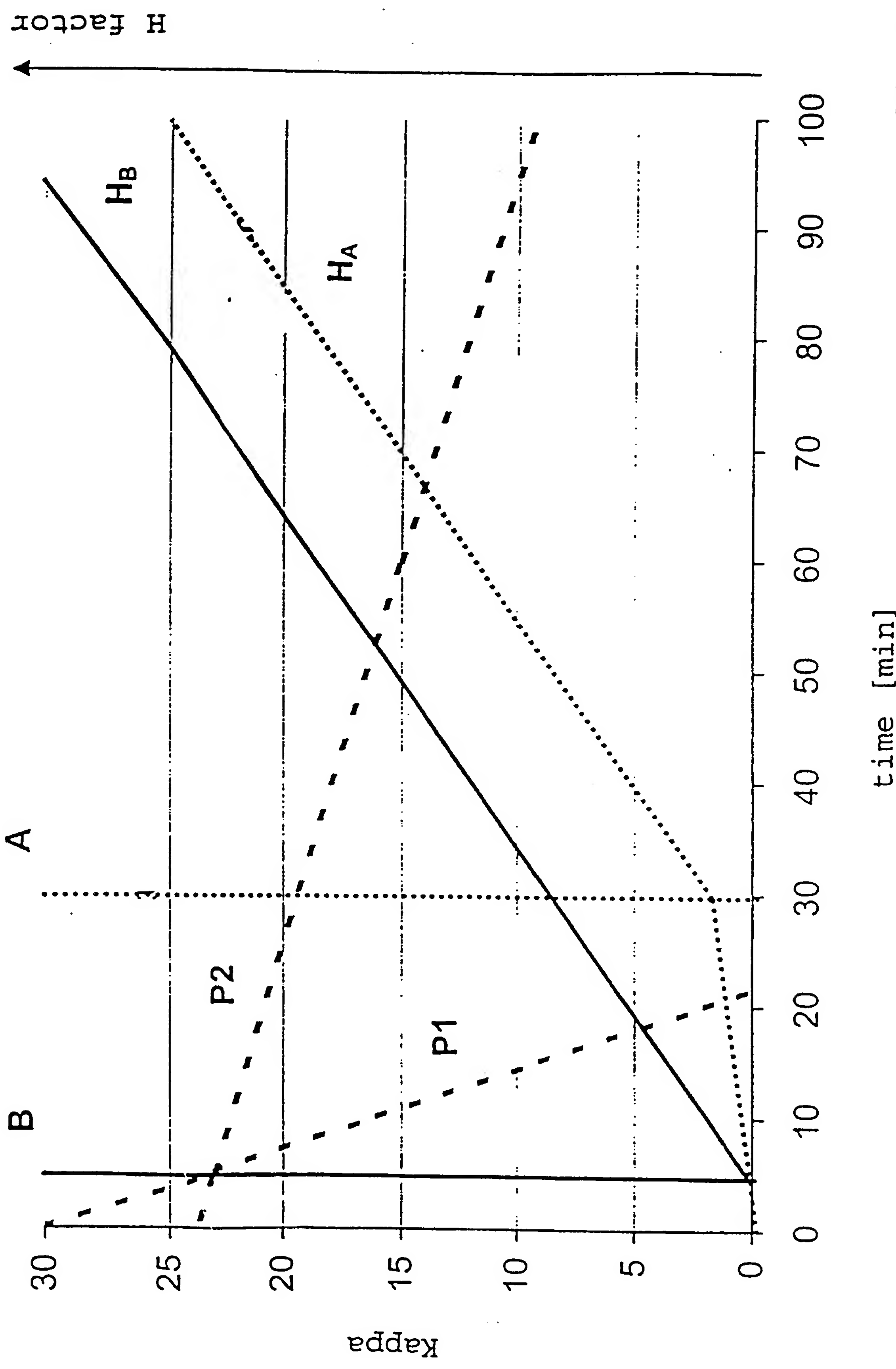
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Fig.2

PATENT CLAIMS

1. System for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, which oxygen delignification takes place in at least two stages and where the system is

c h a r a c t e r i z e d i n that it comprises,

- a first pump (1) which is arranged to pump the pulp to a first mixer (3) for admixing chemicals which are required for the oxygen delignification in this first mixer (3), which first mixer is arranged in close conjunction with the first pump,
- a first delignification zone (6) which is arranged to receive pulp from the first mixer (3),
- a second mixer (8) which is arranged to receive pulp from the first delignification zone, which second mixer comprises means for admixing steam (MP steam) with the pulp,
- a second pump (4) which is arranged to receive pulp after the second mixer,
- a third mixer (5) which is arranged in close conjunction with the second pump, for admixing chemicals which are required for the oxygen delignification in this third mixer (5),
- a second delignification zone (10) which is arranged to receive the pulp from the third mixer.

2. System for oxygen delignification according to Claim 1,

c h a r a c t e r i z e d i n that the first and third mixers (3 and 5, respectively) are mixers using mechanical agitation, with the pulp at least partially being fluidized in gaps in the mixer, and in that the second mixer (8) is a static mixer without mechanical agitation.

3. System for oxygen delignification according to Claim 2,
c h a r a c t e r i z e d i n that it comprises means (O_2) for adding oxygen to the first (3) and third (5) mixers, respectively, and means (MP steam) for adding steam to the second mixer (8).

4. System for oxygen delignification according to Claim 3,
c h a r a c t e r i z e d i n that the second mixer comprises means for supplying steam in a controllable manner (7,V,TC), preferably feedback-controlled in dependence on the temperature of the pulp after the said mixer.

5. System for oxygen delignification according to Claim 4,
c h a r a c t e r i z e d i n that the second mixer (8) consists of a pulp-conveying pipe having a number of inlet holes for the steam in the wall of the pipe.

6. System for oxygen delignification according to Claim 5,
c h a r a c t e r i z e d i n that the steam consists of medium-pressure steam at 8-14 bar.

7. System for oxygen delignification according to Claim 3,
c h a r a c t e r i z e d i n that the system comprises a control system (PC) for controlling the rotational speed of the second pump (4) depending on the pressure in the first delignification zone (6).

8. System for oxygen delignification according to Claim 1,
c h a r a c t e r i z e d

- in that the first delignification zone (6) has a volume which results in a dwell time of 2-20 minutes, preferably 2-10 minutes, and even more advantageously 3-6 minutes, for the pulp in the first delignification zone,

- in that the system is adjusted such that the pressure in the first delignification zone amounts to 0-6 bar, preferably 0-4 bar,

- in that the second pump (4) has a pumping effect such that the pressure in the second delignification zone reaches a level of at least 3 bars overpressure at the top of the second delignification zone, and

- in that the second delignification zone (10) has a volume which is at least 10 times greater than the volume of the first delignification zone, i.e. has a volume which results in a dwell time of at least 20-200 minutes, preferably 20-100 minutes, and even more advantageously within the range 50-90 minutes.

9. Process for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, in at least two stages,

c h a r a c t e r i z e d

a) in that pulp at median concentration is pressurized, and

b) after that, chemicals, chiefly oxygen, are added for the oxygen delignification, such that oxygen delignification takes place in a first stage in which the pulp is treated for a relatively short time, corresponding to 2-20 minutes, preferably 2-10 minutes, and even more advantageously 3-6 minutes, under moderate overpressure within the interval 0-6 bar, preferably 0-4 bar, and at a moderate temperature in the range 85°C ± 10°C,

c) and in that the pulp, after the first stage, and with the pulp being at a median concentration, is

first mixed together with steam for the purpose of increasing the temperature,

d) after which there follows a first pressurization of the heated pulp,

e) and, after that, a second addition of chemicals, chiefly oxygen, for the oxygen delignification,

f) in order, in a concluding stage, to be treated for a longer time than in the first stage, i.e. for a time which is of the order of magnitude of 10 times longer than in the first stage, in the interval 2-200 minutes, preferably 20-100 minutes, and even more advantageously in the interval 50-90 minutes, with this stage taking place at an initial pressure within the interval 8-10 bar, corresponding to the pressure at the inlet of the reactor, but also at a higher temperature, preferably in the range 100°C ± 10°C, but preferably at least 5°C higher than the temperature in the first stage.